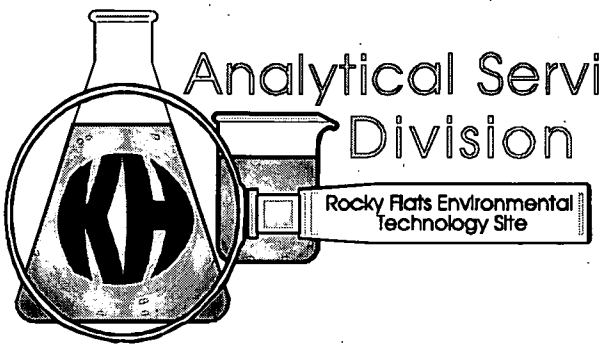

VERIFICATION AND VALIDATION GUIDELINES

FOR

SEMIVOLATILE ORGANICS

DA-SS02-v3

Effective Date: October 1, 2002

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Analytical Services Division

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Date: June 4, 2002

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V & V GUIDELINE CHANGE DESCRIPTION FORM

Instructions: Replace Version 2 with Version 3

Guideline: DA-SS02	**Version:** 3	**Originator:** Ed Brovsky
Description: Verification and Validation Guidelines for Semivolatile Organics.		

Section No.	Change Description
N/A	New version and Effective date
Introduction	A new introduction was written to incorporate the BOA SOW rather than PSA Modules.
Entire Document	For clarity, change bars appearing on a Section Title indicate changes to the entire Section.
Entire Document	References to the BOA SOW and the RFETS BOA Implementation document GR03, are incorporated throughout the document. References to PSA Modules were eliminated. References to Module Specific Verification and Validation (V & V) Guidelines were replaced with Analytical Specific V & V guidelines.
Data Review Checklist	All references to the Data Review Checklist and its examination were removed from the Guidelines.
Entire Document	All actions that involve Reason Codes 801, or 803 were revised to include an NCN be issued to request missing, incomplete data, or corrected data. The action requires the discontinuation of further assessment until corrected data is received and the action also requires a comment in the DQA Report identifying the request for missing or corrected data.
2.14	A section was added for TCLP assessment.

1. PURPOSE AND INTRODUCTION

This document presents those data assessment steps which are unique to Semivolatile Organic Analyses. This Analytical Specific document is to be used in conjunction with DA-GR01, "General Guidelines for data Verification and Validation.

The purpose of this document is to provide guidance in the completion of Data Verification, and Data Validation activities as part of the Rocky Flats Environmental Technology Site (RFETS) Analytical Services Division Data Assessment Process as described in DA-GR01.

This version of DA-SS02 is applicable to Semivolatile Organic Sample Data Packages generated under the National Basic Ordering Agreement (BOA) Statement of Work (SOW) and the Rocky Flats Environmental Technology Site (Site) BOA Implementation Requirements documents, GR03 & GR04.

2. VERIFICATION AND VALIDATION INSTRUCTIONS

The instructions contained in this section are specific to Semivolatiles Organics analyses. They are to be used in conjunction with the general instructions for Verification and Validation found in Analytical Services Division's General Guidelines for Verification and Validation, DA-GR01.

2.1. Chain of Custody, Holding Times, and Sample Preservation

Review Items: COC, Laboratory Sample Receiving Documentation, Cover Page Comments, Sample Case Narrative, raw data, data summary forms, and sample preparation/extraction log.

Objective: The objective is to ascertain the validity of results based on the method required holding times, sample preservation, and the continuity of sample custody.

Source: BOA Attachment 1, § 3.1.2, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Determine if the samples were properly preserved prior to laboratory sample receipt using the criteria provided in Table 1a and Table 1b.

Action 1: If samples were not maintained at 4° ±2° C prior to receipt by the laboratory, do not qualify the sample results. However, comment and assign the reason code **[703]** to all applicable samples.

Item 2: Determine if samples were properly preserved after sample receipt.

Action 2: If documentation specifically indicates sample preservation was not maintained after sample receipt, but prior to analysis, issue a Non-Compliance Notification (NCN) requesting a corrective action to prevent recurrence and qualify all results as estimated **[J 201]**.

Item 3: Determine the actual analysis and preparation holding times by comparing the preparation and analysis dates on the raw data and the sample collection date on the COC. If the actual holding time is greater than the maximum allowable holding time identified in Table 1a or Table 1b, use the following actions to qualify all applicable data:

Action 3a: Qualify all positive results as estimated (J) if the actual holding time was greater than the maximum holding time. Assign code **[J 101]** if the holding time violation is attributed to the laboratory. If the holding time violation is not attributed to the laboratory, assign code **[J 701]**.

Action 3b: Qualify all non-detected results as estimated (UJ) if the actual holding time was greater than the maximum holding time but less than two times the maximum holding time. Assign code **[UJ 101]** if the holding time violation is attributed to the laboratory. If the holding time violation is not attributed to the laboratory, assign code **[UJ 701]**.

Action 3c: Qualify all non-detects as rejected (R) and all detects (J) if the actual holding time was greater than two times the maximum holding time. Assign reason code **[R/J 102]** if the hold time violation is attributed to the lab. If the hold-time violation is not attributed to the laboratory, assign reason code **[R/J 702]**.

Note: Code 701 will apply when samples are received after holding times are expired, or if samples are received after 50% of the holding time has passed.

Table 1a HOLDING TIME AND PRESERVATION CRITERIA

Matrix	Extraction Holding Time (maximum)	Analysis Holding Time (maximum)	Preservation
Water	7 days	40 days	Storage at 4°C,
Soil	14 days	40 days	Storage at 4°C

NOTE: The holding time is based on the date when collection was completed, rather than verified time of sample receipt (VTSR).

Table 1b TCLP EXTRACT HOLDING TIME AND PRESERVATION

Holding Time (Days)			Preservation	
TCLP Extraction	Extract Preparation	Extract Analytical	Non-Aqueous Matrix	Aqueous Matrix
14	14	40	Storage at 4°C	Storage at 4°C

2.2. Sample Data Package Narrative

Review Items: Sample Case Narrative

Objective: Review the narrative for compliance to requirements and for information useful to data assessment.

Source: GR03 § 3.2, BOA Attachment 1, § 3.1.6.2

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Check that the SDP Narrative is present and includes the following as applicable:

- Procedures and/or Standard Method reference for preparation and analysis.
- Descriptions of significant technical difficulties encountered in preparing and analyzing the samples.
- Justification of all dilutions.
- Explanations of any QC deficiencies, missed holding times, or inability to achieve the required detection limits (RDLs).
- Reasons for reanalysis, reanalysis Analytical Batch Identifications Numbers, and a synopsis of the reanalysis Analytical Batch QC Assessment.
- Explanations and descriptions of all deviations from routine protocols, including deviations from approved standard operating procedures (SOPs), detection limit modifications, etc. If it was necessary to contact the CTR for instructions due to the nature of the deviation, the laboratory shall document those instructions in the narrative.

Action 1: If any of the above items are non-compliant, do not qualify the results, comment and include the reason codes **[227]** and/or **[805]** as appropriate. Use professional judgement to determine if the issuance of a NCN is warranted.

2.3. System Monitoring Compound (Surrogate) Recovery

Review Items: Forms 2C/2D, sample preparation/extraction log, sample chromatograms and quantitation reports.

Objective: To assess laboratory performance based on the results of surrogate spike recoveries. Laboratory performance on individual samples is established by means of spiking samples with surrogate compounds prior to extraction and analysis to determine surrogate spike recoveries. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the review and validation of data based on specific sample results are frequently subjective and demand analytical experience and professional judgment.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Check that Forms 2C/2D are present.

Action 1: If forms are missing, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Check that surrogate recoveries are reported for all sample, spike, and blank analyses.

Action 2: If required surrogate recoveries are not provided, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 3: Determine if appropriate reanalysis and reextraction/reanalysis were performed. Reanalysis and reextraction/reanalysis are required for SW-846 if any surrogate recovery is outside the control limits (excludes dilutions). They are required for CLP if more than one base/neutral or acid surrogate is outside the control limits or if any surrogate has a recovery less than 10% (excludes dilutions, sample used for MS/MSD, and surrogates with advisory limits).

Action 3a: If appropriate reanalyses or reextractions were not performed, comment and assign reason code **[142]** to all applicable data.

Action 3b: If appropriate reanalyses/reextractions were performed, determine whether the original analysis or the reanalysis/reextraction is to be reported and provide an explanation in the data quality assessment report.

Item 4: Check that the surrogate percent recoveries (%R) are within the limits of Table 2. If surrogate recoveries for a sample fall outside the control limits, qualify as follows (Specify the fraction that is being qualified (base/neutral or acid):

Action 4a: If the recovery of any two surrogates per fraction are greater than the control limits, estimate **[J 142]** positive results for that fraction. (Determine that high bias was not due to interference with the surrogate compound only.)

Action 4b: If the recovery of any two surrogates per fraction are less than the control limits but greater than or equal to 10%, estimate **[J 142]** positive results and **[UJ 142]** non-detected results for that fraction.

Action 4c: If the recovery of one surrogate is greater than the control limits and the recovery for another surrogate is are less than the control limits but greater than or equal to 10% (in the same fraction), estimate **[J 142]** positive results and **[UJ 142]** non-detected results for that fraction.

Action 4d: If the recovery of any one surrogate is less than 10% in either fraction, estimate **[J 142]** positive results and reject **[R 142]** non-detected results for that fraction.

Table 2 SURROGATE CONTROL LIMITS

Surrogate Compounds	CLP-SOW		SW-846 8270B	
Base/Neutrals	**Water**	**Soil**	**Water**	**Soil**
Nitrobenzene-d5	35-114	23-120	35-114	23-120
2-Fluorobiphenyl	43-116	30-115	43-116	30-115
p-Terphenyl-d14	33-141	18-137	33-141	18-137
1,2-Dichlorobenzene-d4	16-110*	20-130*	--	--
Acids				
Phenol-d5/d6	10-110	24-113	10-94	24-113
2-Fluorophenol	21-110	25-121	21-100	25-121
2,4,6-Tribromophenol	10-123	19-122	10-123	19-122
2-Chlorophenol-d4	33-110*	20-130*	--	--
	*Advisory		(or laboratory control limits)	

Note: Laboratory limits will generally take precedent for SW-846 8270B. Professional judgement may be used to determine the reasonableness of laboratory limits. Limits in Table 2 may be used if laboratory limits are not acceptable.

Dilutions

Compounds reported from the diluted sample will be assessed using the surrogate recoveries from the diluted sample. No action should be taken if a surrogate recovery cannot be reported because of sample dilution. However, professional judgment may be used to warrant qualification.

Item 5: If no surrogate recovery is reported due to dilution, determine if the dilution factor was high enough to justify the surrogates being diluted out.

Action 5: Comment that surrogates were diluted out of the sample and no action was taken. Assign code **[142]** to all sample results associated with diluted surrogates.

Evaluation: ***The following items apply to validation only:***

Item 6: Check chromatograms and quantitation reports to evaluate the recoveries. Verify at least one surrogate recovery per sample.

Action 6: If calculated recoveries are not within 5% of reported result, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue data assessment until a new data package is received.

Item 7: Check raw data for interferences or misidentification when %R values are outside of control limits.

Action 7a: If raw data confirms % R, no action is required.

Action 7b: If raw data indicates misidentification, assign reason code **[804].** Use professional judgment to assign a qualifier based on the severity of the problem.

2.4. MS/MSD Recovery

Review Items: Forms 3C/3Dor equivalent, Forms 6B/6C or equivalent, MS/MSD chromatograms and quantitation reports.

Objective: To determine long-term precision and accuracy of the analytical method on various matrices. These data alone cannot be used to evaluate the precision and accuracy of individual samples.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Check that Forms 3C/3D are present and that MS/MSD analyses were performed at the required frequency.

Action 1: If forms are not present or were not analyzed at the required frequency, comment that the SDP did not include an MS/MSD. No reason code is applied.

Item 2: Check that the MS/MSD percent recoveries (%R) and relative percent differences (RPD), for only the compounds listed in Table 3, are within the identified limits.

Note: No action is taken on MS/MSD data alone to qualify an entire batch. However, using informed professional judgment the data Reviewer may use the MS/MSD results in conjunction with other QC criteria and determine the need for some qualification of data.

Action 2: If MS/MSD recoveries or RPDs are not within the limits of Table 3, comment that limits were not met. Do not qualify, but assign reason code **[231]** to the outlying compound in all associated samples. The data reviewer may use the MS/MSD results in conjunction with other QC criteria to determine if data qualification is warranted.

Table 3 MS/MSD FREQUENCY AND CONTROL LIMITS

Spiking Compound	CLP-SOW %R Limits[RPD Limit]		SW-846 8270* %R Limit
	Water	Soil	Water/Soil
Phenol	12-110[42]	26-90[35]	5-112 or lab limits
2-Chlorophenol	27-123[40]	25-102[50]	23-134 or lab limits
1,4-Dichlorobenzene	36-97[28]	28-104[27]	20-124 or lab limits
N-Nitroso-di-n-propylamine	41-116[38]	41-126[38]	D-230 or lab limits
1,2,4-Trichlorobenzene	39-98[28]	38-107[23]	44-142 or lab limits
4-Chloro-3-methylphenol	23-97[42]	26-103[33]	22-147 or lab limits
Acenaphthene	46-118[31]	31-137[19]	47-145 or lab limit
4-Nitrophenol	10-80[50]	11-114[50]	D-132 or lab limit
2,4-Dinitrotoluene	24-96[38]	28-89[47]	D-191 or lab limit
Pentachlorophenol	9-103[50]	17-109[47]	14-176 or lab limit
Pyrene	26-127[31]	35-142[36]	52-115 or lab limit
	Frequency: 1/20 samples		**Frequency: 1/20 samples**

* SW-846 allows the laboratory to calculate their own recovery limits. Use laboratory limits if provided.

Evaluation: ***The following item applies to validation only:***

Item 3: Calculate at least one percent recovery and one RPD value in the MS/MSD data using the following calculations:

$$\%R = \frac{Found\_Value}{True\_Value} \times 100$$

$$RPD = \frac{|D_1 - D_2|}{\left(\frac{D_1 + D_2}{2}\right)} \times 100$$

where:

D_1 = MS Concentration.

D_2 = MSD Concentration.

Action 3: If the %R or % RPD values cannot be verified to within 5%, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue data assessment until a new data package is received.

2.5. Instrument Performance Check

Review Items: Form 5B or equivalent, decafluorotriphenylphospine (DFTPP) bar graph spectrum, mass listing, and RIC.

Objective: To determine if instrument tuning criteria have been met. Instrument performance checks (tuning) are performed to ensure mass resolution, identification, and to some extent, sensitivity. These criteria are not sample specific. Conformance is established by adherence to acceptance criteria using standard reference materials. These criteria must be met in all circumstances.

Sources: BOA Attachment 1, § 3.2.3; Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Check that Form 5B is present for the all calibrations and that all samples are included.

Action 1: If forms are missing, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Verify that the sample analyses occurred within 12 hours of the daily DFTPP instrument performance check.

Action 2: If the samples were analyzed outside the 12 hour limit, use professional judgment to qualify the data based upon the severity of the problem. At a minimum, comment and assign reason code **[139]** to all applicable data.

Item 3: Check that the DFTPP ion abundance criteria contained in Table 4 are met.

Action 3a: If mass assignment is in error (e.g., m/z 199 is assigned as the base peak), reject **[R 139]** all associated data.

Action 3b: If alternate method criteria are used, comment and assign reason code **[139]**. No additional action is necessary.

Action 3c: If ion abundance criteria are not met, use to following guidelines to qualify data:

- M/z 68, 79, 197, and 441 and the m/z ratios for 198/199 and 442/443 are critical. If any one of these m/z abundances or ratios is not met, reject **[R 139]** all associated data.
- M/z 51, 127, and 275 are not as critical. If any one of these m/z abundances is slightly outside criteria (e.g., m/z 275 at 40% with limits of 10-30%) but the remaining criteria are met, the deficiency is minor. At a minimum, comment and assign reason code **[139]** to all applicable data.
- The relative abundance of m/z 365 indicates suitable instrument zero adjustment. If the relative abundance for m/z 365 is zero, minimum detection limits may be affected. Reject **[R 139]** all applicable data.

- If m/z 365 is present but less than the minimum abundance criteria, the deficiency is not as serious. Use professional judgment to assess the severity of the problem. Either reject **[R 139]** the data or comment and assign reason code **[139]** to all applicable data.

Table 4 DFTPP ION ABUNDANCE CRITERIA

m/z	CLP-SOW	SW-846 8270B
51	30-80% of m/z 198	30-60% of m/z 198
68	<2.0% of m/z 69	<2.0% of m/z 69
69	Mass 69 relative abundance	-- --
70	< 2.0% of mass 69	< 2.0% of mass 69
127	25.0-75.0% of mass 198	40-60% of mass 198
197	<1.0% of mass 198	<1% of mass 198
198	Base Peak, 100% relative abundance	Base Peak, 100% relative abundance
199	5.0 to 9.0% of mass 198	5 to 9% of mass 198
275	10.0-30.0% of mass 198	10-30% of mass 198
365	> 0.75% of mass 198	> 1% of mass 198
441	Present, but < mass 443	Present, but < mass 443
442	40.0-110.0% of mass 198	>40% of mass 198
443	15.0-24.0% of mass 442	17-23% of mass 442

Evaluation: ***The following item applies to validation only:***

Item 4: Verify from the raw data that the mass assignment is correct and that the mass listing is normalized to the correct base peak. Verify that the mass calibration is correct and that there are not transcription errors. Compare the mass listings submitted in the raw data to the reported relative abundances. Recalculate two m/z ratios. If possible, verify that spectra were generated using appropriate background subtraction techniques.

Action 4: If any problems are found, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue data assessment until a new data package is received.

2.6. Internal Standard Area and RT Summary

Review Items: Forms 8B/8C or equivalent, calibration quantitation reports, sample chromatograms and quantitation reports.

Objective: To determine if the GC/MS sensitivity and response are stable. Internal standard (IS) performance criteria ensure that the GC/MS sensitivity and response are stable for every analytical run.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Check that Forms 8B/8C are present for all calibrations associated with sample analyses and that all samples and blanks are included.

Action 1: If form(s) are missing, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Determine if the IS compounds vary from the following required/recommended IS compounds: 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10; phenanthrene-d10, chrysene-d12, and perylene-d12. CLP specifies these compounds; SW-846 recommends them.

Action 2: If the IS compounds vary from the method, use professional judgment to assess the impact on the data.

Item 3: The IS area count in the samples and blanks must not differ by more than a factor of two (-50% to +100%) from the area count measured in the associated calibration standard. If so, qualify as follows:

Action 3a: If the area count for any IS in a sample is above the acceptance limits (+100%), estimate **[J 143]** positive results for compounds quantitated using that IS.

Action 3b If the area count for any IS in a sample is below the acceptance limits (-50%), estimate **[J 143]** positive results and **[UJ 143]** non-detected results for compounds quantitated using that IS.

Action 3c If the area count for any IS in a sample is extremely low (i.e., less than 50% of the lower control limit), or if instrument sensitivity exhibits a major abrupt drop off, reject **[R 143]** non-detected results and estimate **[J 143]** positive results for compounds quantitated using that IS.

Evaluation: ***The following item applies to verification only:***

Item 4: Determine if the IS retention time in the continuing calibration standard varies by more than ±30 seconds from the last daily calibration standard.

Action 4: If ±30 seconds is exceeded, reject **[R 143]** affected non-detected results and estimate **[J 143]** affected positive results for compounds quantitated using that IS.

Evaluation: ***The following items apply to validation only:***

Item 5: Verify that area counts and retention times are correctly transcribed from the sample and standard quantitation reports onto the Forms 8B/8C.

Action 5: Depending upon the magnitude of the problem, issue a NCN to request clarification or explanation of the data. Comment and assign reason code

[804] to all applicable data. Alternatively, use the times and area counts from the quantitation reports.

Item 6: Determine if the IS retention time in the continuing calibration standard varies by more than ±30 seconds from the last daily calibration standard.

Action 6: If so, examine the chromatogram for evidence of false positives or false negatives. Use professional judgment to qualify the data based upon the chromatogram and magnitude of the shift. If qualification is warranted, reject **[R 143]** affected non-detected results and estimate **[J 143]** affected positive results for compounds quantitated using that IS.

2.7. **Sample Results** (Target Identification)

Review Items: Form 1B/1C or equivalent, Forms 6B/6C or equivalent, Forms 7B/7C or equivalent, COC record, sample extraction/preparation logs, and sample chromatograms and quantitation reports.

Objective: To determine if false positives (reporting a compound present when it is not) or false negatives (not reporting a compound that is present) were reported by evaluating qualitative criteria for compound identification.

Sources: Attachment 1 to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Check that Forms 1B/1C is present for each sample.

Action 1: If forms are missing, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Check that significant figures and flagging protocol are as specified in the latest version of CLP.

Action 2: If significant problems exist, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 3: Determine if Forms 1B/1C contain "B" qualifiers.

Action 3a: If "B" qualifiers are present, determine if bank contamination is addressed in the SDP Narrative. If contamination is not addressed, do not qualify the results. Comment and include the reason code **[805].**

Action 3b: If "B" qualifiers are present, proceed with the qualification specified under Blanks.

Evaluation: ***The following items apply to validation only:***

Item 4: Verify that all significant peaks on the chromatogram are accounted for as target compounds, TICs, surrogates, and internal standards.

Action 4a: In the event a target compound is misidentified, correct the specific data point on the Form 1 (initial and date) and assign the reason code **[251].**

Action 4b: In the event a target compound was misidentified and the Form 1 was manually changed, the CTR or their designee shall be informed in writing of potential EDD discrepancies this change may cause.

Relative Retention Time

Item 5: Confirm positive results by reviewing the relative retention times (RRTs) for positive results on the sample integration reports to those for the associated calibration standard. Verify the RRTs are within ±0.06 RRT units of the standard RRT.

$$RRT = \left(\frac{RT_{com}}{RT_{is}} \right)$$

where:

RT_{com} = Retention time of the compound of interest

RT_{is} = Retention time of nearest internal standard

Action 5: If the RRT results are not within ±0.06 RRT units of the standard RRT qualify the associated result as estimated **[J 145]**.

Item 6: Determine if the mass spectra of the sample compound and a laboratory-generated standard meet the following:

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- The characteristic ions from the reference mass spectrum (the three ions of greatest intensity or any ions over 30% relative intensity if fewer than three such ions occur) maximize within one scan of those in the sample mass spectrum.
- The relative intensities of these ions must agree within ±30% between the sample and standard spectrum.
- Structural isomers (very similar mass spectra) should be identified separately if the height of the valley between the isomer peaks is less than 25% of the sum of the two peaks. Otherwise, structural isomers are identified as isomeric pairs.

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- All ions present in the standard mass spectrum at a relative intensity greater than 10% must be present in the sample spectrum.
- The relative intensities of these ions must agree within ±20% between the sample and standard spectrum.
- Ions present at greater than 10% in the sample spectrum but not present in the standard spectrum must be considered (reviewed for possible background contamination or presence of coeluting compounds) and accounted for.

Note: The application of qualitative criteria for GC/MS analysis compounds requires professional judgment.

Action 6a: If it is determined that an incorrect false positive exists, the result should be reported as non-detected **[U 145]**.

Action 6b: If it is determined that a false negative exists, reject **[R 145]** the non-detected result if the unreported positive result exceeds the RDL.

Note: Be aware of situations (e.g., high concentration samples preceding low concentration samples) when sample carry-over is a possibility. Use professional judgment to determine if carry-over occurred and that the data was qualified appropriately.

2.8. Compound Quantitation and RDL

Review Items: Form 1B/1C or equivalent, Forms 6B/6C or equivalent, COC record, sample preparation/extraction logs, sample chromatograms and quantitation reports.

Objective: To ensure that the reported quantitation results and detection limits are accurate.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Using the Line Item Code from the COC record, determine if the detection limits reported on Form 1B/1C match the required detection limits (RDLs) listed in Attachment K to BOA Attachment 1, GR03, GR04, or other applicable Statement of Work (SOW). Note that dilutions, percent solids, and extraction steps will impact the final RDLs reported.

If RDLs on Form 1B/1C do not meet those required by the Line Item Code requested, check the RIN file for additional information, which may explain the deviation.

Action 1: If an explanation is not found, use professional judgement to qualify non-detected results with reason code **[213]**.

Item 2: Evaluate Forms 1B/1C to ensure that no "E" qualifiers are present. If "E" qualifiers are present, ensure that another Form 1B/1C with a diluted sample analysis is present in the data package.

Action 2: If "E" qualifiers are present and there is not a Form 1B/1C with a diluted sample analysis, comment and estimate **[J 148]** the positive "E" result.

Note: Generally, the analysis with the lower reporting limits are used with the exception of results that exceed the calibration range. Only compounds that originally exceeded the calibration range are reported from the dilution.

Item 3: Ensure that required dilutions are addressed in the SDP Narrative.

Action 3: If not addressed, do not qualify the results. Comment and include the reason code **[805]**.

Item 4: Determine from the Form 1B/1C the compounds that were outside the upper half of the calibration range prior to dilution, but fall within the upper half of the calibration range after dilution.

Action 4: Assign reason code **[155]** only to the data points that meet Item 4 criteria. Do not assign any qualifier to these data points. Any data qualification will be assigned to the data point reported from the dilution.

Item 5: Determine if the diluted sample analysis keeps the response of the major constituents in the upper half of the calibration range.

Action 5: If the diluted sample analysis fails to keep the response of the major constituents in the upper half of the calibration range, use professional judgment to qualify the data. At a minimum, comment and assign reason code **[252]** to all applicable data.

Evaluation: ***The following items apply to validation only:***

Item 6 Verify that compound quantitation, as well as the adjustment of the MDLs, is performed according to the method. Target compound quantitation should be performed using the internal standard associated by the method.

Action 6: If not compliant, use professional judgment to qualify the data.

Item 7: Ensure that the same internal standard was used in the standard as in the sample.

Action 7: If a different internal standard was used in the sample than in the standard, do not qualify any data. Issue a NCN to request corrected data, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 8 Target compounds should be quantitated from the primary ion listed in the method unless the primary ion has interference from the sample.

If a secondary quantitation ion was used, verify that the standard was also quantitated from the same secondary quantitation ion.

Action 8: If a different quantitation ion was used in the sample than in the standard, do not qualify any data. Issue a NCN to request the corrected data, comment, and assign reason code **[804]** to all applicable data.

Item 9: Verify that acceptable (NIST/EPA/MSDC) Mass Spectral Library Searches are provided for all sample identifications.

Action 9a: If reference spectra are not provided, but applicable reference spectra can be found elsewhere in the data package, comment and issue a NCN and assign the reason code **[802]** to all applicable data.

Action 9b: If acceptable reference spectra cannot be found, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into

the NCN. Discontinue the data assessment until a new data package is received.

Item 10 Verify that all B and E qualifiers are explained in the case narrative.

Action 10: If not, comment and assign the reason code **[805]** to all applicable data.

Calculations

Item 11: Examine the raw data to verify the correct calculation of one positive result per sample. Quantitation reports, chromatograms, sample preparation/extraction logs, dilutions, and cleanups are compared to the reported sample results.

Calculate using the following equations:

Water and water-miscible waste:

$$\frac{ug}{L} = \frac{A_X \times I_S \times V_t \times DF}{A_{is} \times RRF \times V_0 \times V_i}$$

where:

A_X = Response of the characteristic ion for the analyte in the sample, area counts.

I_S = Amount of internal standard injected, ng.

V_t = Volume of total extract, uL.

DF = Dilution factor.

A_{is} = Response of the characteristic ion for the internal standard, area counts.

RRF = Response factor for the analyte from the appropriate calibration standard.

V_0 = Volume of water purged, mL.

V_i = Volume of extract injected, uL.

Sediment/soil, sludge, and waste:

$$\frac{ug}{Kg} = \frac{A_X \times I_S \times V_t \times DF}{A_{is} \times RRF \times V_i \times W_S \times P}$$

where:

A_X, I_S, A_{is}, RRF = Same as in water and water-miscible waste above.

V_t = Volume of total extract, uL.

DF = Dilution factor.

V_i = Volume of extract injected, uL.

W_S = Weight of sample extract, g.

P = Percent Solids/100.

TICs only:

A_x = Total response of the TIC in the sample, area counts.

A_{is} = Total response of the nearest internal standard free of interference, area counts.

RRF = One (1.0).

Action 11a: If significant problems exist, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Action 11b: If the RRF values are not verified within 5%, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.9. CLP Tentatively Identified Compounds (TICs)

Review Items: Form 1F or equivalent, sample extraction/preparation logs, and sample chromatograms and quantitation reports.

Objective: To determine if TICs were qualitatively identified. Peaks not identified as target compounds, surrogates, or internal standards are potential TICs. TICs must be qualitatively identified by a National Institute of Standards and Technology (NIST) mass spectral library search and the identifications assessed by the data reviewer.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Check that Form 1F is present for each sample.

Action 1: If forms are missing, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Verify that all TIC results are qualified by the laboratories as "**NJ**".

Action 2: If TIC results are not qualified as "**NJ**" by the laboratory, "**NJ**" shall be applied to all applicable results by the data assessor with reason code **[804]**.

Evaluation: ***The following items apply to validation only:***

Item 3: Verify that the alkane series are properly identified and reported as series (e.g., C5-C9 as a single entry along with the estimate for the total concentration of the series).

Action 3: If non compliant, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 4: Verify that no semivolatile target compounds are mistakenly reported as TICs.

Action 4: If target compounds are mistakenly reported as TICs, the quantitation is in error due to the use of total area. Issue a NCN, comment and assign reason code **[226]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 5: Verify that volatile compounds reported as target analytes in the volatile fraction are not reported as TICs in the semivolatile fraction.

Action 5: If non-compliant, reject **[R 199]** the TIC.

Note: If a TIC result in the sample is not sufficiently above the level in the blank, the TIC result should not be reported. (Generally, the 10X blank rule is applied here.)

Item 6: Verify that the laboratory has generated a NIST/EPA/MSDC mass spectral library search for all required peaks in the chromatograms for samples and blanks.

Action 6a: If non-compliant, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Action 6b: When a compound is not found in any blanks but is a suspected common laboratory artifact/contaminant, reject **[R 199]** the TIC. Examples:

- Common contaminants: CO2 (m/z 44), siloxanes (m/z 73), diethyl ether, hexane, certain freons (1,1,2-trichloro-1, 2,2-trifluoroethane or fluorotrichloromethane), and phthalates at less than 100ug/L or 4000 ug/Kg.
- Solvent preservative cyclohexene (methylene chloride preservative). Related by-products cyclohexanone, cyclohexenone, cyclohexanol, cyclohexenol, chlorocyclohexene, and chlorocyclohexanol.
- Aldol reaction products of acetone including: 4-hydroxy-4-methy-2-pentanone, 4-methyl-2-penten-2-one, and 5,5-dimethyl-2 (5H)-furanone.

Item 7: Evaluate the spectra using the following guidance:

- All ions present in the standard mass spectrum at a relative intensity greater than 10% should be present in the sample spectrum.
- The relative intensities of these ions must agree within ±20% between the sample and reference spectrum.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not present in the reference spectrum must be reviewed for possible background contamination, interference, or coelution of additional TIC or target compounds.

Action 7a: When the above criteria are not met but the identification is correct in the technical judgment of the data reviewer or mass spectral interpretation specialist, report the TIC as is.

Action 7b: If the identification is uncertain in the data reviewer's judgment, or there are extenuating factors affecting compound identification, the TIC result may be reported as "unknown" or changed to an appropriate identification.

Action 7c: If more than one possible match exists, the TIC may be reported as "either compound X or compound Y."

Action 7d: If isomer specificity is in question, the TIC result may be changed to a non-specific result (e.g., 1,3,5-trimethyl benzene to trimethyl benzene isomer).

Action 7e: If identification is uncertain but other samples have a TIC of similar RRT and the same ions, identification information may be inferred.

Item 8: Examine blank chromatograms to verify that sample TICs are not found in the blanks.

Note: Be aware that TICs at low levels in the samples may also be present in the blanks at levels below 10% of the internal standard height. This is particularly likely for common laboratory artifacts and contaminants.

Action 8: If a TIC identification is changed for any of the above reasons, add the reason code **[226]** to all affected data points.

Item 9: Examine the raw data to verify the correct calculation of one TIC result per sample. Use the equation in Section 2.8.

Action 9: If significant problems exist, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.10. Calibration

Review Items: Forms 6B/6Cor equivalent, Forms 7B/7C or equivalent, sample and standard chromatograms and quantitation reports.

Objective: To determine if the instrument calibration is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run and of producing a linear calibration curve. Continuing calibration establishes the 12-hour relative response factors on which the quantitations are based and checks satisfactory performance of the instrument on a day-to-day basis.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Initial Calibration

Item 1: Determine if the initial calibration includes all target compounds and surrogates, is performed within 12 hours of the associated instrument performance check, and is performed at the beginning of the sequence or when continuing calibration criteria are not met. The acceptance criteria used for samples are as contained in Table 5.

Table 5 INITIAL CALIBRATION CRITERIA

Method	No. of Standards	Concentration	RRF Limit	%RSD Limit	Correlation Coefficient or r^2
CLP	5	20, 50, 80, 120, and 160 ng/uL	RRF > 0.05	30%	N/A
SW-846 8270	5	Low near but above established MDL; others should define the range of the expected concentrations	RRF > 0.05	30%	0.99

Action 1a: If an inappropriate number of standards or inappropriate concentration levels are analyzed, use professional judgment to assess the impact on the data. At a minimum, comment and assign reason code **[168]** to all applicable data.

Action 1b: Estimate **[J 140]** positive results and **[UJ 140]** non-detected results for those compounds whose %RSDs or linearity exceed the criteria in the associated initial calibration.

Action 1c: Estimate **[J 140]** positive results for those compounds whose RRFs are less than 0.05.

Action 1d: Reject **[R 140]** non-detected results for those compounds whose RRFs are less than 0.05.

Continuing Calibration

Item 2: Determine if the continuing calibration includes all target compounds and surrogates, and is analyzed at the beginning of each 12-hour analysis period following the instrument performance check and prior to the sample and blank analyses. The acceptance criteria used for samples are contained in Table 6.

Table 6 CONTINUING CALIBRATION CRITERIA

Method	Concentration	RRF Limit	%D Limit
CLP	50 ng/uL	RRF > 0.05	25%
SW-846 8270	Mid-level	RRF > 0.05	25%

Action 2a: If the continuing calibration frequency criteria are not met or if inappropriate concentration levels are analyzed, use professional judgment to assess the impact on the data. At a minimum, comment and assign reason code **[168]** to all applicable data.

Action 2b: Estimate **[J 141]** positive results and **[UJ 141]** non-detected results for those compounds whose %Ds exceed the criteria in the continuing calibration.

Action 2c: Estimate **[J 141]** positive results for those compounds whose RRFs are less than 0.05.

Action 2d: Reject **[R 141]** non-detected results for those compounds whose RRFs are less than 0.05.

Evaluation: ***The following items apply to validation only:***

Initial Calibration

Item 3: Determine if the target compounds are quantitated from the primary ion listed in the method unless the primary ion has interference from the sample.

Note: If a secondary quantitation ion was used, verify that the standard was also quantitated from the same secondary quantitation ion.

Action 3: If a different quantitation ion was used in the sample than in the standard, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 4: Check the raw data and verify at least one RRF per calibration standard. Recalculate at least one average RRF and %RSD:

$$RRF = \frac{A_X \times C_{is}}{A_{is} \times C_X}$$

where:

A_X = Response for the characteristic ion for the analyte to be measured, units area counts.

C_{is} = Concentration of the internal standard, ug/L.

A_{is} = Response for the characteristic ion for the internal standard, units area counts.

C_X = Concentration of the analyte to be measured, ug/L.

$$\%RSD = \frac{SD}{\overline{X}} \times 100$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(X_i - \overline{X}\right)^2}{(n-1)}}$$

where:

X_i = Each individual value used to calculate the mean

$\overline{X}$ = The mean of n values

n = The total number of values.

Action 4: If the RRF for the %RSD cannot be verified to within 0.5%, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Continuing Calibration

Item 5: Determine if target compounds are quantitated from the primary ion listed in the method unless the primary ion has interference from the sample.

Note: If a secondary quantitation ion was used, verify that the standard was also quantitated from the same secondary quantitation ion.

Action 5: If a different quantitation ion was used in the sample than in the standard, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 6: Recalculate at least one daily RRF and %D:

$$\%D = \frac{R_1 - R_2}{R_1} \times 100$$

where:

R_1 = Calibration factor from first analysis.

R_2 = Calibration factor from subsequent analysis.

Action 6: If calculated results are not verifiable to within 5%, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.11. Quality Control Check Samples (SW-846)

Review Items: Form 3C or equivalent

Objective: To evaluate the results of the quality control check sample to determine the accuracy of the analytical method and laboratory performance.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and verification:***

Item 1: Check that Form 3C is present.

Action 1: If a form 3C is missing, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

QC Check Sample Frequency

Item 2: Determine if one QC Check sample is analyzed with each batch of samples processed within a working shift (up to 20 samples).

Action 2a: If the frequency requirement is not met, do not qualify the data, however, assign reason code **[168]** to the data.

Action 2b: If a QC Check sample was not analyzed, qualify all results as **[J 168]**.

QC Check Sample Percent Recovery

Item 3: Check that the QC Check sample percent recoveries (%R) for only the compounds in Table 7 are within the following limits:

Table 7 QUALITY CONTROL SAMPLE PERCENT RECOVERY LIMITS

Spiking Compound	%R Limit
Phenol	5-112
2-Chlorophenol	23-134
1,4-Dichlorobenzene	20-124
N-Nitroso-di-n-propylamine	D-230
1,2,4-Trichlorobenzene	44-142
4-Chloro-3-methylphenol	22-147
Acenaphthene	47-145
4-Nitrophenol	D-132
2,4-Dinitrotoluene	D-191
Pentachlorophenol	14-176
Pyrene	52-115
	(or may use laboratory limits)

Action 3: No action is taken on quality control sample data alone to qualify an entire batch. However, using informed professional judgment the data Reviewer may use the quality control sample results in conjunction with other QC criteria and determine the need for some qualification of data. Do not qualify the data. However, assign the reason code **[110]**.

Evaluation: ***The following item applies to validation only:***

Item 4: Calculate at least one percent recovery in the quality control sample data using the following calculation:

$$\%R = \frac{Found\_Value}{True\_Value} \times 100$$

Action 4: If calculated results are not verifiable to within 5%, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.12. Blanks

Review Items: Form 4B or equivalent, Method Blank Forms 1B/1C or equivalent, chromatograms and quantitation reports.

Objective: To assess the laboratory blank analysis results to determine the existence and magnitude of contamination problems. The criteria for evaluation of laboratory blanks apply to any blank associated with the samples (e.g., method, instrument, trip, or equipment blanks). If problems with any blank exist, all data associated with the method blank must be carefully evaluated to determine whether or not there is an inherent variability in the data or if the problem is an isolated occurrence not affecting other data.

Sources: Attachment I to BOA Attachment 1, and Base Method

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Verify that Method Blank Summary Forms (4A) are present.

Action 1: If not provided, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Check that Forms 1B/1C is present for each blank.

Action 2: If not provided, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 3: Check that Form 1F is present for each blank.

Action 3: If not provided, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 4: Determine if the blank criteria contained in Table 8 are compliant for the given method.

Note: If more than one blank is associated with a sample, qualification should be based upon comparison of the blank with the highest level of contamination.

Table 8: BLANK CRITERIA

Method	Frequency	Criteria
CLP	Per matrix, per concentration level, for each extraction batch (not to exceed 20 samples)	No contaminants should be present in the blanks. The method blanks should be analyzed on each GC/MS system used to analyze that set of associated samples.
SW-846 8270	A method blank should be extracted with each extraction batch, when there is a change in reagents, and following any concentrated sample that has saturated ions from a compound.	No interference at or above the MDL. The blank samples should be carried through all stages of the sample preparation and measurement steps (i.e., the method blank should be analyzed on the same instrument as the samples).

Action 4a: If the proper blanks were not analyzed at the appropriate frequency, determine the severity of the problem and its effect on the data using professional judgment. At a minimum, comment and assign reason code **[168]** to all applicable data.

Action 4b: If a target compound is found at any concentration in the blanks but not in the samples, no action is taken.

Action 4c: If a target compound is found in the blanks at any concentration and is also found in the sample, apply the following:

- If the sample concentration is less than 5 times the blank concentration (10 times for common contaminants) and less than or equal to the RDL, qualify the data as estimated **[JB 249]** (EDD results cannot be raised to the RDL).
- If the sample concentration is less than or equal to 5 times the blank concentration (10 times for common contaminants) and greater than the RDL, qualify the data **[U249]**.
- If the sample concentration is greater than 5 (10 times for common contaminants) times the blank concentration and greater than the RDL do not qualify the reported value.

Note 1: The Reviewer must consider the weights, volumes, percent solids, and dilution factors when applying the 5x and 10x rules. These factors must be accounted for so that an actual

comparison of the contamination is made. The Reviewer should be particularly aware of sample results which undiluted exceed the action level, but fall within the action level as a result of the subsequent dilution.

Note 2: The common contaminants according to the National Functional Guidelines for Organic Data Review are:

Semivolatile Extractables:

◊ Phthalate Esters

- If an associated method blank exhibits gross contamination, reject **[R 249]** positive results for the affected compounds.

  Note: The Functional Guidelines define gross contamination as saturated peaks. Professional judgment must be used to assess the impact the contamination has on the associated samples and which compounds are considered affected.

Action 4d: If an associated method blank was not analyzed for the samples, estimate **[J 249]** positive results.

Evaluation: ***The following items applies to validation only:***

Item 5: Verify that all significant peaks on the chromatogram are accounted for as target compounds, TICs, surrogates, and internal standards.

Action 5a: In the event a target compound, surrogate, or internal standard are misidentified, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package and EDD is received.

Action 5b: In the event a TIC is misidentified, comment in the Data Quality Assessment Report.

Item 6: Recalculate one positive result per blank. Review the chromatograms and quantitation reports to evaluate blank results.

Action 6: If the calculated result does not agree within 5% or if a compound was misidentified, comment and assign reason code **[804]** to all applicable data. Review all other positive blank results.

2.13. Sample Preparation Raw Data

Review Items: Raw Data

Objective: To check that sample preparation raw data deliverable requirements have been met and that raw data are present in a form suitable for data assessment.

Sources: Attachment I to BOA Attachment 1, Base Methods

Evaluation: ***The following items apply to validation activities only:***

Item 1: Check that preparation raw data (benchsheets and/or preparation logs) are included for all analyses performed and include the following:

- Analytical Batch identifier
- Date of preparation
- Identifiers for all samples, sample duplicates, and spikes
- Identifiers for at least one preparation blank and lab control sample
- For aqueous samples initial and final volumes for all samples and QC samples
- For solids and non-aqueous liquids reported by weight, initial weights and final volumes for all samples and QC samples
- For samples reported by weight, balance identifiers with dates of use.
- Dated signatures for at least one analyst and one reviewer

Action 1a: Check this item as complete if raw data were sufficient to perform calculations for all previous items.

Action 1b: Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code **[804].** An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 1c: For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Verify that instrument run logs are available for all analytical sequences.

Action 2a: Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code **[804].** An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 2b: For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 3: If GPC is performed, verify sample results are adjusted to account for the GPC cleanup.

Action 3: If sample results are not adjusted to account for GPC cleanup, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.14. TCLP Sample and Extract Preparation (Summary Form 2)

Review Items: Form 2 or equivalent, and raw data.

Objectives: To determine if samples were evaluated and prepared by the proper TCLP preparation method according to LIC, analyte, sample matrix, and analytical method utilized.

Sources: Attachment I to BOA Attachment 1, GR03 § 5, and Method 1311 for TCLP extraction.

Evaluation: ***The following Items apply to both verification and validation:***

Item 1: Check that a Form 2 or equivalent is present and the following information is included:

- Lab name, Lab Code, Analytical Batch Identifier and the RIN.
- Form 2 data for each sample.
- Physical descriptions of the samples (e.g. *multiphase liquid*, or *solids with no free liquid*) and a statement about which samples are of the same matrix.
- Result for the preliminary determination of percent solids and a description of the method of determination.
- An indication of whether particle size reduction was completed and how the reduction was completed, if reduction was required.
- A *Yes* or *No* to indicate whether free liquid was present in the sample.
- A *Yes, No, or N/A* to indicate whether any free liquid present was miscible with the extraction fluid.
- A volume recorded if a non-miscible liquid is present.
- A check that the preliminary evaluation of the pH of solids is recorded.
- A check that the evaluation of the pH of solids after the addition of acid is recorded, if applicable.
- A *Net Sample Weight (g)* or total weight of sample taken for the extraction process is recorded.
- A *Net Weight of Solids Extracted (g)* or the net weight of solids remaining after liquid solid separation is recorded.
- The type and weight of the extraction fluid added to the extraction vessel is recorded.
- The *Date and Time* of the start and end of the extraction period were recorded.
- The pH for the leachate solution after extraction and filtration, but before preservation was recorded.
- The method of preservation of the leachate was recorded.
- At least one spike-sample was prepared per waste type and analytical batch.
- At least one extraction blank was prepared per extraction fluid type and analytical batch.
- At least one duplicate sample was prepared per waste type and analytical batch.

Action 1a: Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code **[804].** An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 1b: For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Evaluation: ***The following items apply to validation only:***

Item 2: Determine that the appropriate TCLP Extraction method was completed for each sample.

Action 2: If the incorrect method was used for sample preparation and a CTR approved deviation was not documented, estimate **[J 207]** all applicable data.

Item 3: Check for evidence that samples with solids less than 0.5% were filtered as a TCLP Extract.

Action 3: If the percent solids is less than 0.5% and the sample was not filtered, estimate **[J 220]** positive results that exceed the regulatory level.

Item 4: Check for evidence of particle size reduction when the sample particle size exceeds 9.5 mm or the surface area is less than $3.1 cm^2$.

Action 4: If particle size reduction is required and reduction was not performed, estimate **[J 222]** all sample results less than the regulatory level.

Item 5: Verify that TCLP results for extracts of samples with free liquids, both miscible and non-miscible, were reported appropriately.

Action 5a: If a single combined TCLP result was not reported for a sample with both miscible and non-miscible liquids and this deviation was not addressed in the narrative, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Action 5b: If a single combined TCLP result was not reported for a sample with both miscible and non-miscible liquids and this deviation was addressed in the narrative, comment and assign the reason code **[248].**

Item 6: Verify that the correct Extraction Fluid Type was used for the TCLP according to the following:.

- If the pH before or after (as applicable) the acidification is less than 5, Extraction Fluid Type 1 is to be used for the TCLP of all analyses.
- If the pH after acidification is greater than 5, Extraction Fluid Type 2 is to be used for the TCLP of all analyses.
- Extraction Fluid Type 1 is to have a pH of 4.93 ± 0.05
- Extraction Fluid Type 2 is to have a pH of 2.88 ± 0.05

Action 6a: If an incorrect or improperly prepared Extraction Fluid Type was used for the TCLP, comment and qualify using professional judgment, but qualify at a minimum as estimated **[J 233].**

Action 6b: If the extraction fluids are not numbered and cannot be identified from the data, comment and qualify using professional judgment, but qualify at a minimum as estimated **[J 224].**

Item 7: Verify that the correct amount of sample was processed for the TCLP.

Action 7: If the net sample weight processed for TCLP is less than 100 grams, use professional judgment to determine if the sample size is too small. Consider the physical state of the sample, the availability of sample, potential mixed waste issues (waste minimization priority), and whether particle size reduction was performed. At a minimum, comment and assign the reason code **[123].**

Item 8: Verify that the extraction period was within 16 to 20 hours.

Action 8: If the extraction start and end dates and times are not available or if the extraction time is not within 16-20 hours, use professional judgment to evaluate the data. Results near the regulatory limit may be biased low if the extraction time is less than 16 hours and results just above the regulatory limit may be biased high if the extraction time is greater than 20 hours. Results just below the regulatory limit that are suspected of low bias due to an insufficiently short extraction time are Rejected **[R 225].**

Item 9: Verify that TCLP Extracts were preserved appropriately, if analysis was not completed immediately.

Action 9: If the TCLP Extracts were not analyzed immediately after extraction and were not preserved at 4 ± 2° C after extraction, comment and qualify all results less than the regulatory limit as estimated **[J 201].**

Item 10: Verify that a minimum of one TCLP Spike, Blank, and Duplicate are processed per waste type, preparation batch and extraction fluid type.

Action 10: If evidence of a spiked sample, duplicate sample, or extraction blank are not provided, comment and qualify all results as rejected **[R 168].**

Item 11: Verify that the ambient temperature during the extraction was maintained at 23 ± 2° C.

Action 11: If the ambient temperature during TCLP extraction was not maintained at 23 ± 2° C, estimate **[J 201]** all results less than the regulatory limit.

3. DATA QUALITY ASSESSMENT REPORT PREPARATION

Prepare a Data Quality Assessment Report according to the General Data Assessment guidelines presented in DA-GR01. A Data Quality Assessment Report template for DV-SS02 is presented as Attachment 1.

4. REFERENCES

- USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, October 1999.
- Reason Codes for Data Assessment, Analytical Services Document
- RFETS BOA Implementation Requirements, GR03 Version A.5
- RFETS BOA Implementation Requirements, GR04 Version A
- Basic Ordering Agreement (BOA) for Laboratory Analytical Services administered by Westinghouse Savannah River Company on behalf of the Department of Energy.

ATTACHMENT 1: DATA QUALITY ASSESSMENT REPORT TEMPLATE

SVO

Data Quality Assessment Report
Rocky Flats Environmental Technology Site

RIN Number	Analytical Method/Analytical Specific Line Item Code		Review Level
Analytical Laboratory	**Assessment Performed by**	**Data Assessment Guideline Identifiers**	**Number of Samples**

Sample Numbers: _____

Quality Control Items	Reviewed (Y or N)	Non-Compliance Identified
General (Cover Page, Narrative)		
Chain of Custody		
Holding Times		
Sample Preservation		
Surrogate Recovery		
Matrix Spike/Matrix Spike Duplicate		
Instrument Performance Check		
Internal Standards		
Sample Results		
Tentatively Identified Compounds		
Calibration		
Quality Control Check Samples		
Blanks		
Sample Preparation		
EDD		
Other:		

Y Item was reviewed or non-compliance was identified
N Item was not reviewed or non-compliance was not identified
N/A Item is not applicable to the Line Item

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Data Quality Assessment Report

Rocky Flats Environmental Technology Site

Data Assessment results are classified as either Action Items or Comments. Action Items are technical non-compliances that result in qualification of analytical results. Data may be qualified as valid (V), estimated (J), presumptively estimated (NJ), estimated at an elevated level of detection (UJ), or rejected (R). Multiple qualifiers may be associated with any given data point based on the number of problems identified, however, the assigned qualifier is based upon the following hierarchy: R, UJ, NJ, J, V. All data points that are not qualified based upon action items in this report are considered valid (V). Comments are technical non-compliances or contractual non-compliances that do not result in qualification of data.

Action Items:

Comments:

Verification/Validation Signature_____ Date:_____

Reviewer Signature_____ Date:_____
(*Validation Only*)